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厦门大学

博士学位论文

激光解析/电离飞行时间质谱机理探讨及应用

Laser Desorption/Ionization Time-of-Flight Mass Spectrometry: Mechanism and Application

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专业名称：分析化学

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摘 要

相对于溶液分析，固体直接分析技术具有省时省力，避免繁琐的样品前处理，避免污染物引入及样品损失等的风险，同时保留了样品成分的空间分布和深度分布等信息。因此，在原位、快速、实时、无损、低消耗等分析需求日渐增长的工业及科学界，固体分析意义尤为突出。发展一种准确可靠的高通量、高选择性及高灵敏度的固体直接分析技术是我们一直追求的目标。

激光自发明以来，因为其出色的物理特性，稳定的性能，操作上的可控性和高度重现性而广泛用于分析科学领域。质谱作为一种现代的分析仪器，其优越的定性能力和高灵敏度的检测能力而备受瞩目。因此，激光与质谱的结合成为必然。激光作为质谱离子源，它提供一种通用的采样和离子化手段。在低能量下，激光作为一种软电离源，其发展经历了最早的激光解析（Laser desorption）用于有机固体分析到现在不断成熟的基质辅助激光解析电离（Matrix-assisted laser desorption ionization, MALDI）用于聚合物或生物大分子分析。在高能量下，激光电离（Laser ionization, LI）提供一种用于元素分析最通用的原子化、激发及电离方法。尽管激光与质谱联用技术广泛用于固体分析，但是对激光离子源的认识仍然需要深入研究，特别是激光与物质作用机理、离子形成及等离子体膨胀等方面。所以本论文主要利用飞行时间质谱（Time-of-flight mass spectrometry, TOFMS）开展以下三个方面的工作：

1．在高激光功率密度下利用激光溅射垂直引入式飞行时间质谱（LI-O-TOFMS）研究激光溅射等离子体羽传输特性，特别是在离子源有背景气体（He或Ar）参与的情况下。通过离子动能测定，考察等离子体羽膨胀动力学及离子与背景气体相互作用行为。我们发现了等离子体羽分裂（Plume splitting）现象，其传输特性从真空下的自由膨胀到相对低背景气压下的冲击波型流体动力学膨胀过渡直至最后相对高背景气压下的完全热能化扩散。

2．在低激光功率密度下关注 MALDI-MS分析中的一个有趣现象，就是谱图中的大部分离子价态是一价。我们首次提出三体碰撞复合（Three-body recombination）机理作为离子降价的原因。电子被认为在离子价态还原中起到了至关重要的作用

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。我们发现基于电子密度和电子温度的三体复合速率常数比两体复合要高很多倍，且离子价态越高越容易复合，所以谱图中一价离子占主导主要是因为它被还原成中性粒子的几率最小。我们讨论了电子的来源，并通过四个实验证据来支持三体碰撞复合理论。

3．从实际固体样品分析上展示激光与质谱联用技术的强大。我们利用LI-O-TOFMS分析生物样品，只需简单的样品处理，就能对黄花鱼卵单细胞进行元素的定性及半定量分析。LI-O-TOFMS获得的谱图信噪比高，无论是金属还是非金属元素都清晰可见且同位素比合理。LI - O-TOFMS与ICP-MS分析结果非常接近。出色的定性及无标样半定量分析能力表明我们仪器在固体直接分析上的优越性能。

关键词：固体直接分析；激光-物质作用；等离子体羽膨胀；基质辅助激光解析

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Abstract

In contrast to solution analysis, the direct solid analysis has many advantages, e.g. little or even no sample size requirement and sample preparation, low risk of reagent or solution waste, the avoidable introduction of contamination, and high spatial (lateral and in -depth) resolution. In particular, the demands of real - time, in-situ, fast and non-destructive analysis have become much more important in the fields of industry and science. Thus, it is alw ays the goal for us to develop reliable analytical methods with high throughput, high selectivity and high sensitivity. Laser has been widely used in analytical field for its stability, controllability, reproducibility and excellent physical property. Mass spectrometry is one of the most promising techniques for its capabilities of sensitive, precise, and accurate

multielemental determinations as well as isotope ratio measurements. The combination of laser and mass spectrometry is very powerful. As an ion s ource, laser provides an universal technique for solid sampling and ionization. In the low energy regime, laser desorption is used for organic solid analysis and matrix - assisted laser desorption ionization (MALDI) is especially suitable for the analysis of polymer and

biomolecule. In the high energy regime, laser ionization (LI) is best for elemental composition analysis with atomization, excitation and ionization at one step. Although the combination of laser and mass spectrometry is widely used for solid analysis, the mechanisms involved in the laser-solid interaction , plasma formation and plume expansion are still not fully understood. Thus, in this dissertation, researches are carried out by the using of time-of-flight mass spectrometry (TOFMS). Related works are briefly described as follows:

1.In high laser irradiance, c haracteristics of laser ionization in vacuum and low pressur e background gas (He or Ar) have been investigated through LI-O-

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TOFMS. The measurement of kinetic energy and spatial distributions of ions was made to illustrate the plume expansion dynamics and the interaction with background gas. plume splitting was observed in the low pressure regime investigated (from 100 Pa to 2000 Pa). The plume propagation translates from a free expansion in vacuum to shockwave-like expansion at relative low pressure and finally thermalized diffusion into background gas at relative high pressure environment.

2.In low laser irradiance, we focused on one aspect of MALDI source. Ions in MALDI are predominantly singly charged for small analyte molecules with insufficient degrees of freedom. With the estimated high number density and low temperature of electrons, the three-body recombination mechanism is attractive and should be considered as an important caus e for the charge reduction in the plume.The higher of the charge state, the more efficient of reduction. The predominantly singly charged ions in mass spectrum may primarily attribute to their lower probability of neutralization. Theoretical calculations indicate that the three -body recombination will overtake the two-body recombination. There are four experimental proofs supporting our theory.

3.Finally, LI- O-TOFMS was employed to determine the elements in single egg cell. The sample preparation procedure was simple and fast by just heating the corvina egg cells at a high temperature. In the LI-O-TOFMS system, elements can be clearly observed in the spectrum with reasonable isotope ratios. Some of the nonmetallic elements, such as P and S that are difficult to be measured by ICPMS, can also be determined by LI-O-TOFMS.The results demonstrate that LI-O-TOFMS is capable of elemental quantification of a single cell of several hundreds micrometer in size.

Keywords: Solid direct analysis; Laser-solid interaction; Plume expansion; MALDI;

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